Physicochemical Characterization and Gas Sensing Studies of $Cr_{1-x}Fe_xNbO_4$ and Application of Principal Component Analysis

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Thesis Abstract

Monitoring the working environment of laboratories and industries for pollutants is of primary concern to ensure the healthiness of working personnel. Semiconducting metal oxides (SMOs) are sensitive to the gas ambience and can be tuned for sensing purpose. As SMOs are not selective, an array of sensors with differential selectivity may resolve to great extent. The objective of the thesis is to understand the physicochemical properties and gas sensing characteristics of $Cr_{1-x}Fe_xNbO_4$. Applying principal component analysis to the sensor response data either for selection of features or for differentiation of analytes is also of concern.

Preparation of $Cr_{1-x}Fe_xNbO_4$, phase characterization, lattice parameters estimation, morphological and micro chemical analysis (SEM & EDX), electrical characterization by direct current (DC & AC) in the temperature range of 423 K to 573 K, weighted magnetic moment of iron and chromium deduced from susceptibility measurements, spin nature of iron and surface compositions of different valences of chromium and iron deduced from X-ray photoelectron spectroscopy of are presented. The wide dynamic range hydrogen sensing characteristics of $CrNbO_4$ bulk pellets at different temperatures along with the cross-sensitivity towards NH_3, $NO_x(NO+NO_2)$ and PG (petroleum gas) are investigated. The preparation of $Cr_{1-x}Fe_xNbO_4$ thick and thin films by screen-printing and PLD are also presented. The thick films are tested at different temperatures towards hydrogen. The n-type or p-type nature of thick films towards hydrogen with varying iron concentration in $Cr_{1-x}Fe_xNbO_4$ is reported. The thin films are characterized for phase formation, morphology by XRD, SEM and AFM. XPS performed surface characterization. Electrical resistance measurements at different temperatures and preliminary experiments on hydrogen sensing are presented. The probable hydrogen sensing mechanism of $CrNbO_4$ was revealed by X-ray photoelectron spectroscopy. The experimentally observed reduction in metal ion oxidation states upon interacting with hydrogen is best illustrated by Kröger-Vink notation.

Principal component analysis was applied for three different types of studies: i) The fit parameters of the transient response of $CrNbO_4$ thick films towards hydrogen are analyzed for finding out the better feature for calibration, ii) Different thick films of $CrNbO_4$, $Cr_{0.5}Fe_{0.5}NbO_4$ and $FeNbO_4$ operated at various temperatures for testing H_2 and VOCs are analyzed for redundancy in sensor behavior and iii) $Cr_{0.8}Fe_{0.2}NbO_4$ thick films are studied for sensing H_2, NH_3 and their mixtures and usefulness of PCA in resolving them in PC-space. In addition, H_2 and VOCs are tested at different temperatures and redundancy in temperature is deduced to construct a sensor array with a minimum number of sensors. Finally, a sensor array consisting of $Cr_{0.8}Fe_{0.2}NbO_4$ thick films, operating at different temperatures is built, and qualitative discrimination of analytes in PC-space is demonstrated. Finally, the major findings of the present investigations and suggestions for future aspects of experimentation are provided